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(पहला पुनरीक्षण)

Indian Standard

METALLIC MATERIALS — DETERMINATION OF LINEAR THERMAL EXPANSION

(First Revision)

UDC 669 : 536·413·2

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BUREAU OF INDIAN STANDARDS
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Mechanical Testing of Metals Sectional Committee, MTD 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Mechanical Testing of Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The test method described in this standard, is a reliable method of determining the linear thermal expansion of metallic materials, which are widely used in electronic devices, compensating pendulums, balance wheels, automobile pistons and bimetal strips, etc. This method may also be applied to ceramics, refractories, glasses, rocks, minerals, plastics, woods, cements, pastes, mortars, and composites. However, many materials and certain material applications require detailed preconditioning and specific thermal test schedules for correct evaluation of thermal expansion. Since a general test method can not cover all specific requirements, details of this nature should be contained in particular material specifications.

This standard was first published in 1965. In this revision following modifications have been made:

a) Temperature range of test has been modified and now the test method is applicable over the range of "—180°C to +900°C" instead of temperature up to +1 000°C.

b) Definition of coefficient of linear thermal expansion has been modified.

c) Vitreous silica dilatometer has replaced Quartz tube dilatometer.

d) Clauses on computer data analysis and precision and bias have been incorporated.

In the revision of this standard, assistance has been derived from ASTM E 228 — 85 'Standarrd Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer' issued by the American Society for Testing and Materials, USA.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METALLIC MATERIALS — DETERMINATION OF LINEAR THERMAL EXPANSION

(First Revision)

1 SCOPE

This standard describes the method for determination of linear thermal expansion of metallic materials over the temperature range of −180 to 900°C using vitreous silica dilatometers.

2 TERMINOLOGY

2.0 For the purpose of this standard, the definitions given below shall apply.

2.1 Initial Diameter

Initial diameter of the specimen is the diameter of the test specimen measured at room temperature before the test is started.

2.2 Initial Length

Initial length of the specimen is the length of the test specimen measured at room temperature before the test is started but after the thermocouples have been attached. This is necessary in order to avoid errors due to any change caused by the method of attaching the thermocouples.

2.3 Linear Thermal Expansion

The change in length per unit length resulting from a temperature change. Linear thermal expansion is symbolically represented by $\Delta L/L_0$, where ΔL is the observed change in length ($L_2 - L_1$), and L_0, L_1, and L_2 are the lengths of the specimen at reference temperature T_0 and test temperatures T_1 and T_2. Linear thermal expansion is often expressed as a percentage or in parts per million (such as µm/m).

2.3.1 *Instantaneous Coefficient of Linear Thermal Expansion*

The slope of the linear thermal expansion curve at temperature T. Instantaneous coefficient of linear thermal expansion is represented by :

$$\alpha_T = (1/L_0)\, dL/dT$$

The value of the instantaneous coefficient must be accompanied by the temperature at which it is determined.

2.3.2 *Mean Coefficient of Linear Thermal Expansion*

The linear thermal expansion per change in temperature. The mean coefficient of linear thermal expansion represented by:

$$\bar{\alpha} = [(1/L_0) (\Delta L/\Delta T)]$$
$$= (1/L_0) [(L_2 - L_1)/(T_2 - T_1)]$$

The value of the mean coefficient must be accompanied by the values of the two temperatures.

2.4 Vitreous Silica Dilatometer

A device that measures the difference in linear thermal expansion between a test specimen and the vitreous silica parts of the dilatometer.

3 SUMMARY OF METHOD

3.1 This method uses a vitreous silica dilatometer of either the tube or the rod type to determine the change in length of a solid material as a function of temperature.

3.2 The temperature can be controlled either over a series of steps or at a constant heating or cooling rate over the entire range.

3.3 The linear thermal expansion and the coefficients of linear thermal expansion can be calculated from the recorded data.

4 APPARATUS

4.1 Dilatometer, consisting of

4.1.1 Specimen holder or probes or both, made of vitreous silica. See **5.8.2** for recommended annealing schedule of the vitreous silica.

4.1.2 Furnace, cryostat, and bath, used for uniformly heating or cooling the specimen over the temperature range of interest but not below −180°C or above 900°C. The temperatures shall be controlled as a function of time.

4.1.3 Transducer (a dial gauge or differential transformer is commonly used) for measuring the difference in length between the specimen and the specimen holder or probe with an accuracy of at least ± 1·0 µm. The transducer shall be protected or mounted so that the maximum temperature change observed in the transducer during a test will affect the transducer readings by less than 1·0 µm.

4.1.4 Temperature sensor, for measuring the mean temperature of the specimen with an accuracy of at least ± 0·5°C. If a thermocouple is used, it shall be referenced to the ice point with an ice-water bath or an equivalent system.

4.2 Caliper, for measuring the reference length L_0, of the specimen with an accuracy of at least ± 25 µm.

5 PRECAUTIONS

5.1 Heating vitreous silica above 800°C may cause viscous flow and a time-dependent change in its thermal expansion. The magnitude of these effects will depend on the particular type of vitreous silica.

5.2 Heating vitreous silica, that is contaminated with alkali compounds, above 500°C may cause it to crystallize. To avoid this, it should be cleaned by immersion for 1 min in an aqueous solution containing 10% hydrofluoric acid followed by a thorough rinse with distilled water. The vitreous silica should not be touched with the hands after cleaning to avoid contamination with alkali compounds.

5.3 Inelastic creep of a specimen at elevated temperatures can often be avoided by making its cross section sufficiently large.

5.4 Care may be needed to control the temperature gradient in long specimens.

5.5 Moisture should be kept out of the dilatometer, especially when used at cryogenic temperatures.

5.6 A closed specimen holder may be needed when the dilatometer is immersed in a liquid, for example, a bath.

5.7 The specimen should be supported or held in a position so that it is stable during the test.

5.8 The specimen holder or probes, or both, must be made of the same type of vitreous silica.

5.8.1 A test run with a vitreous silica specimen should not result in an apparent mean coefficient of linear thermal expansion larger than ± 0·3 µm/m°C, when applying the system calibration outlined in **8**.

5.8.2 Annealing the vitreous silica specimen and dilatometer parts in the following steps may reduce any excessive lot-to-lot differences in linear thermal expansion.

5.8.2.1 Heat at 100°C/h to 1 200°C

5.8.2.2 Hold at 1 200°C for 2 h

5.8.2.3 Cool at 60°C/h to 1 000°C

5.8.2.4 Cool at 120°C/h to 600°C

5.8.2.5 Cool at 200°C/h to room temperature.

5.9 Since the precision and accuracy of the length measurements are fixed for a specific apparatus, to obtain the same percent uncertainty in the coefficient, a much larger temperature range must be used for low expansion materials than for high expansion materials. Conversely, if the same temperature range is used, the percent uncertainty of the coefficient will be much larger for the low expansion material.

6 TEST SPECIMENS

6.1 The specimen length L_0, shall be such that the accuracy of determining $\Delta L/L_0$ will be at least ± 20 µm/m.

NOTE — For example, if the transducer is accurate to ± 2 µm then the length must be at least 01. m.

6.2 The end surfaces of the specimen (as well as the contacting surface of the specimen holder, or rods, or both) should not be rougher than about 10 µm RMS and shall be uniform in shape. Pointed specimen ends, that will deform during a test, shall be avoided.

7 CALIBRATION

7.1 Calibrate the transducer by imposing a series of known displacements with a precision screw micrometer, end-gauge blocks, or equally accurate device.

7.2 Calibrate the temperature sensor.

7.3 The dilatometer, as a total system, shall be calibrated by measuring at least one reference material of known thermal expansion. Recommended reference materials are listed in Annex A. A reference material with expansion as close as possible to the sample material should be selected.

7.3.1 Either one of the following two calibration constants indicated in **9.1** may be used:

$$A = 1/(\Delta L/L_0)m\,[(\Delta L/L_0)t - (\Delta L/L_0)s]$$

or

$$B = (\Delta L/L_0)t - (\Delta L/L_0)m$$

where

m = the measured expansion of the reference material,

t = the true or certified expansion of the reference material,

s = the assumed or known expansion of the vitreous silica parts of the dilatometer, and

A, B = the function of the temperature.

7.3.2 The calibration should be done using the same test procedure as that used for the test specimen; for example, the same sample length and temperature schedule.

8 PROCEDURE

8.0 Each step may not need to be followed in the sequence given and, depending upon the apparatus, all steps may not be needed.

8.1 Measure the initial (room temperature) length of the specimen, record as length L_0.

8.2 Place the specimen in the dilatometer after making certain that all contacting surfaces are free of foreign material. Take care to ensure good seating of the specimen in a stable position.

8.3 Place the temperature sensor at the mid-length of the specimen. Preferably it should be in intimate contact with the specimen; but it shall at least be as near the specimen as possible. The temperature sensor shall not restrict movement of the specimen in the dilatometer.

8.4 Make certain that the transducer is in stable contact with the probe(s) and specimen.

8.5 Place the assembled dilatometer into the furnace cryostat or bath, and allow the temperature of the specimen to come to equilibrium.

8.6 Record the initial readings of the temperature sensor, T_1 and the transducer L_1.

8.7 Heat or cool the specimen to the desired temperature according to the following procedures.

8.7.1 Hold at a series of constant temperatures until the transducer reading reaches a constant value (variation < ±2·0 μm). At that point the indicated temperature of the specimen shall not vary by more than ±2°C and the temperature gradient in the specimen shall not exceed 0·5°C per cm. See Annex A for recommended construction details. Hold time is a function of the thermal mass of the dilatometer and specimens and will vary for different temperatures. Readings of temperature, T_1 and changed specimen length, L_1 need to be recorded at each constant temperature T_1.

8.7.2 Heat or cool at some constant rate that is less than 3°C per min. When using this procedure the mean temperature of the specimen will likely be different from the measured temperature (lower on heating and higher on cooling) but if the system is correctly calibrated with a reference material the measured expansion of the test specimen will be accurate. Readings of temperature and change of length should be continuously recorded.

8.7.3 If the length of the specimen at the end of the test is different by more than 20 μm/m a retest should be considered or this permanent deformation taken into accord when reporting the expansion values.

9 CALCULATION

9.1 Depending on which calibration constant used, calculate the linear thermal expansion of the test specimen as follows:

$$(\triangle L/L_0) = A(\triangle L/L_0) + (\triangle L/L_0)s$$

or

$$(\triangle L/L_0) = (\triangle L/L_0) + B$$

where

$(\triangle L/L_0)$ = the expansion as indicated by the transducer,

s = the assumed or known expansion of the vitreous silica parts of the dilatometer, and

A, B = constants determined as shown in **7.3.1**.

9.2 Using the calculated values of the linear thermal expansion, calculate the mean coefficients by dividing by the appropriate temperature range:

$$\bar{\alpha} = [(\triangle L/L_0)/\triangle T]$$

9.3 The following instantaneons coefficients, are determined from the slopes. dL/dT, of the length versus temperature curve at each appropriate temperature. The slope may be determined either graphically from a plot or numerically from an equation fit to the data:

$$\alpha_T = (1/L_0)\, dL/dT$$

10 COMPUTER DATA ANALYSIS

Computer or electronic-based instruments, techniques, or data treatment equivalent to this test method may also be used. Users of this test method are specially advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to determine the necessary equivalency prior to use. In the case of dispute, only the manual procedures described in this test method are to be considered valid.

11 TEST REPORT

11.0 The report may include the following information if available and applicable.

11.1 Description of material, including name of manufacturer, chemical composition, thermal and mechanical history, moisture content, and other conditioning history.

11.2 Method of preparation of test specimen, form and dimensions, and axial orientation if the material is anisotropic.

11.3 Brief description of the apparatus including displacement and temperature measuring systems, estimate of precision, heating and cooling rates, temperature controls, and any protective gas other than air.

11.4 Listing of reference material(s) and procedure used to calibrate the dilatometer system including the transducer and temperature sensor.

11.5 Tabulation of data including initial length (L_0) at reference temperature (T_a) showing test temperature and linear thermal expansion.

11.6 The following tabulation or curves as required:

$$\frac{\triangle L}{L_0} \text{ versus } T : \bar{\alpha} \text{ versus T: } \alpha_T \text{ versus } T$$

where the actual temperatures used to calculate are listed.

11.7 Complete description of any usual behaviour of the specimen, such as a permanent change in length, excessive oxidation, scaling, discoloration, deformation, cracking, or spalling, that may be of value in interpreting test results.

11.8 Any additional information required by a particular material specification.

12 PRECISION AND BIAS

12.1 Precision and bias of determining both thermal expansion and the mean coefficient of thermal expansion depend upon the simultaneous measurement of temperature and relative length.

12.2 Random error is usually associated with precision and bias of repeated length and temperature measurements, but other variables may also intrude on the measurements. For instance, a specimen may change position repeatably or the voltage applied to the transducer may fluctuate.

12.3 Systematic error is usually larger and can result from many sources. These include the accuracy of the length and temperature measurements, the deviation of the specimen mean temperature from that indicated by the sensor, the deviation from linearity of the transducer, the temperature gradients between the specimen holder or probes, or both, the difference between the assumed or measured value of expansion of the vitreous silica, and the effect of additional surface contacts between the specimen and the transducer. Little can be done to improve the random errors once the transducer and temperature sensor have been selected except to follow good experimental practice. Systematic errors, however, can be reduced by careful calibration of the individual components and of the total system.

ANNEX A

(*Clause* 7.3)

RECOMMENDED REFERENCE MATERIALS

Temperature	*Linear Thermal Expansion*	
	Platinum	Aluminium
(°C)	(μm/m)	(μm/m)
−195	−1 756·65	0
−150	−1 420·60	−3 430
−100	−1 024·09	−2 550
− 50	− 607·96	−1 550
0	− 176·20	− 460
20	0	0
50	268·06	710
100	722·38	1 900
200	1 654·60	4450
300	2 612·01	7 130
400	3 592·18	10 050
500	4 596·55	13 230
600	5 628·65	16 760
700	6 692·81	—
800	7 793·27	—

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